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# OPTIMUM RANGES FOR X-RAY THICKNESS MEASUREMENTS

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OPTIMUM RANGES FOR X-RAY THICKNESS MEASUREMENTS

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As illustrated in Figure 1, film thicknesses can be measured by two x-ray methods:

1) X-ray absorption (gauging or radiography). The incident beam passes through the material, and the attenuation of the beam by the material is measured.

2) X-ray fluorescence. If the material consists of elements which fluoresce in the accessible region of the x-ray spectrum, the intensity of that fluorescence is related to thickness. There are three possibilities.

   a) The fluorescence of a material behind the specimen can be measured. This is analogous to (1) above.

   b) Source and detector are both on the same side of the specimen.

   c) Source and detector are on opposite sides of the specimen.

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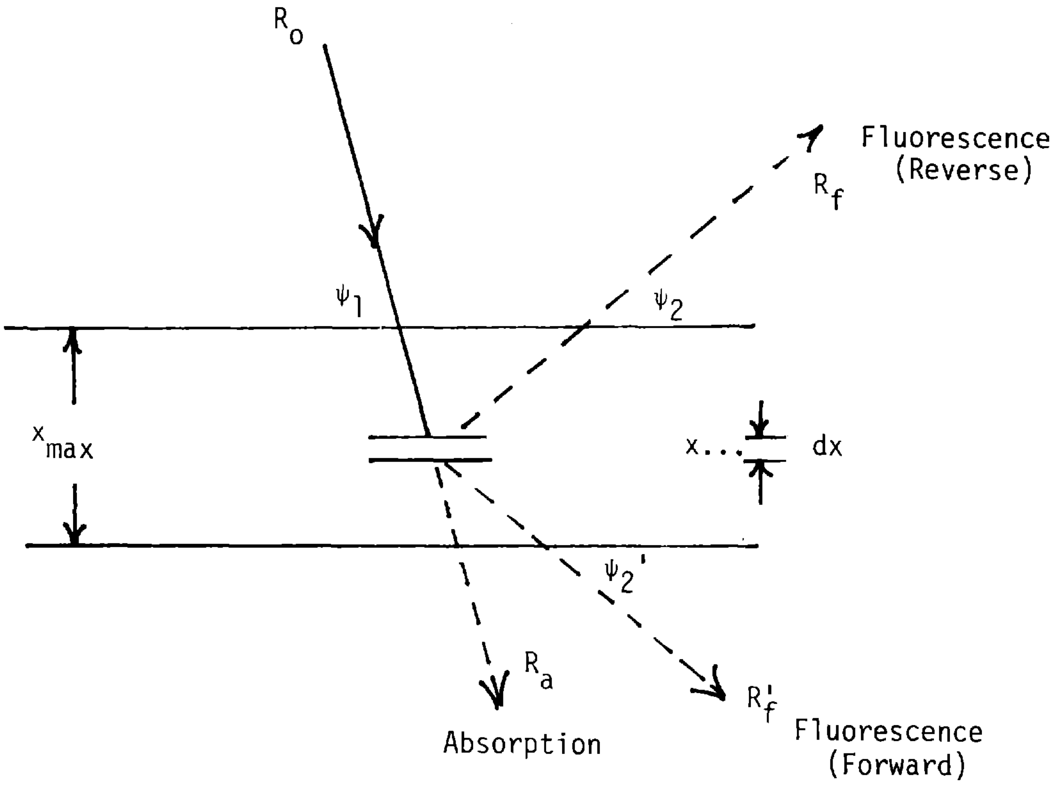


Figure 1. Thickness measurement by absorption ($R_a$), by fluorescence in the back direction ($R_f$), and by fluorescence in the forward direction ($R_f'$).

A rule-of-thumb used in absorption measurements is that the best precision is obtained when the incident beam is attenuated by $e^{-2}$ (that is, $\mu\rho x = 2$). Questions which arise are:

1) What are the analogous rule-of-thumb for fluorescence measurements (forward and reverse directions)?

2) What happens to precision at non-optimum thickness? Over what ranges are the methods best applied?

The answers are summarized in the table below. The remainder of this note provides the methodology and equations used to obtain the results.

| | Optimum $\mu\rho x$ | Minimum error when $N_{max}=10^5$ | Range of $\mu\rho x$ to double error | Range for Gold* |
|---|---|---|---|---|
| Absorption | 2.0 | .43% | .46-5.4 | 7.9-93 $mg/cm^2$ |
| Fluorescence-Rev. | .64 | .64% | .067-2.3 | 0.37-13 $mg/cm^2$ |
| Fluorescence-For. | .57 | .67% | .067-1.5 | 0.37-8.3 $mg/cm^2$ |

*Assuming silver incident radiation

$\mu_{AgK\alpha,\ Au} = 58$

$\mu_{AuL\alpha,\ Au} = 123$

90° geometry

## Sensitivity to Change in Areal Density

The equations for absorption and fluorescence are very similar. In fact, the curves in figure 2 are mirror images of each other.

Absorption: $^{R}/R_{o} = e^{-\mu\rho x}$

Fluorescence: $^{R}/R_{o} = 1 - e^{-\mu\rho x}$

where R = counting rate from the specimen

$R_o$ = maximum counting rate. For absorption, this is the incident beam intensity (i.e., no specimen).

For fluorescence, the maximum rate is obtained at "infinite" thickness.

We want a large change in relative counting rate per unit change in areal density so that we can distinguish between small increments of areal density.

The sensitivity is found by differentiations:

Absorption : $$\frac{d(^{R}/R_{o})}{d(\rho x)} = -\mu e^{-\mu\rho x}$$

Fluorescence : $$\frac{d(^{R}/R_{o})}{d(\rho x)} = \mu e^{-\mu\rho x}$$

We see from these equations (and from Figure 2), that the greatest sensitivity is when the areal density approaches zero (that is,

$d(^R/R_0)/d(\rho x)$ approaches $|\mu|$. Conversely, the sensitivity approaches zero as the thickness increases toward "infinity.".

Clearly, the area to be avoided is that of thick specimens where the sensitivity approaches zero. One might conclude that the converse is true, that the best measurements are made with thin specimens, where the sensitivity in the measured parameter $(^R/R_0)$ is greatest. Further reflection indicates this idea is not valid.

When the specimen is very thin, there is very little to measure. In the fluorescence case, the counting rate is small and the counting statistics are poor. With absorption, the counting rate is at its highest, but the error in the number of counts represents a large <u>relative</u> error in the areal density.

We therefore conclude that there is an optimum range of thicknesses for measurement, somewhere between the extremes of very thin and very thick specimens. What follows is the calculation of the optimum ranges.

<u>Equations for Fluorescence in the Forward and Reverse Directions:</u>

(Refer to Figure 1)

1) The primary beam is attenuated passing through the material to the infinitesimal dx: $\mathrm{Attn}_1 = (e^{-\mu_1 \rho x})$

2) At dx, fluorescence occurs: $dI = I_o^{'}K'C\rho dx$

3) The fluorescent beam is attenuated as it passes out of the material: $Attn_2(rev) = e^{-\mu_2 \rho x}$

$$Attn_2(for) = e^{-\mu_2 \rho (x_{max} - x)}$$

where $\mu_1$ = mass abs. coef. for the primary radiation, corrected for path length (csc $\psi_1$)

$\mu_2$ = mass abs. coef. for the fluorescent radiation, corrected for path length (csc $\psi_2$)

K' = sensitivity factor, usually emperically determined from a standard material

C = weight fraction of the fluorescent element

$\rho$ = density

x = thickness

The fluorescent intensity is found by combining the factors and integrating over the thickness of the material:

$$dI = I_o Attn_1 \times Attn_2 \times KC\rho dx$$

$$\text{Reverse:} \int_0^R dI = I_o K'C\rho \int_0^{x_{max}} e^{-(\mu_1 + \mu_2)\rho x} dx$$

$$R = \frac{KC}{\mu_1 + \mu_2}\left(1 - e^{-(\mu_1 + \mu_2)\rho x_{max}}\right)$$

$$R = R_o \left(1 - e^{-\mu\rho x_{max}}\right)$$

where $\mu = \mu_1 + \mu_2$

$R_o$ = intensity at "infinite" thickness $= \frac{KC}{\mu}$

Forward: $dI = I_o K'C\rho\, e^{-\mu_1 \rho x}\, e^{-\mu_2 (x_{max} - x)\rho}\, dx$

$$\int_o^R dI = I_o K'C\rho\, e^{-\mu_2 \rho x_{max}} \int_o^{x_{max}} e^{-(\mu_1 - \mu_2)\rho x}\, dx$$

$$R = \frac{K'CI_o}{(\mu_1 - \mu_2)}\, e^{-\mu_2 \rho x_{max}}\left(1 - e^{-(\mu_1 - \mu_2)\rho x_{max}}\right)$$

$$R = \frac{KC}{(\mu_1 - \mu_2)}\left(e^{-\mu_2 \rho x_{max}} - e^{-\mu_1 \rho x_{max}}\right)$$

In limiting cases, the two equations become identical:

1) $\mu_1 \to o \quad R_{for} \to R_{rev} = \frac{KC}{\mu_2}\left(1 - e^{-\mu_2 \rho x}\right)$

2) $\rho x \to o \quad R_{for} \to R_{rev} = KC\, \rho x$

The functional relationships between relative intensities and areal densities are plotted in Figure 2.

Find the minimum in measurement error as a function of areal density:

We seek the fractional error in the areal density measurement as a function of the number of counts in some given time. Once we know the functional relationship, we can find its minimum, and thus the optimum areal density for measurement.

1) Absorption: Basic eqn: $^R/R_o = e^{-\mu\rho x}$

The counting rate is: $R = {}^N/t$

So that: $\frac{N}{R_o t} = e^{-\mu\rho x}$

Solving for areal density: $(\rho x) = -\frac{1}{\mu} \cdot \ln\left(\frac{N}{R_o t}\right)$

$$= -\frac{1}{\mu}\left\{\ln(N) - \ln(R_o t)\right\}$$

Find the sensitivity of areal density to changes in the numbers of counts:

$$\frac{d(\rho x)}{dN} = -\frac{1}{\mu} \cdot \frac{1}{N}$$

$$d(\rho x) = -\frac{1}{\mu} \cdot \frac{1}{N} \cdot dN$$

INTENSITY - THICKNESS PROFILES

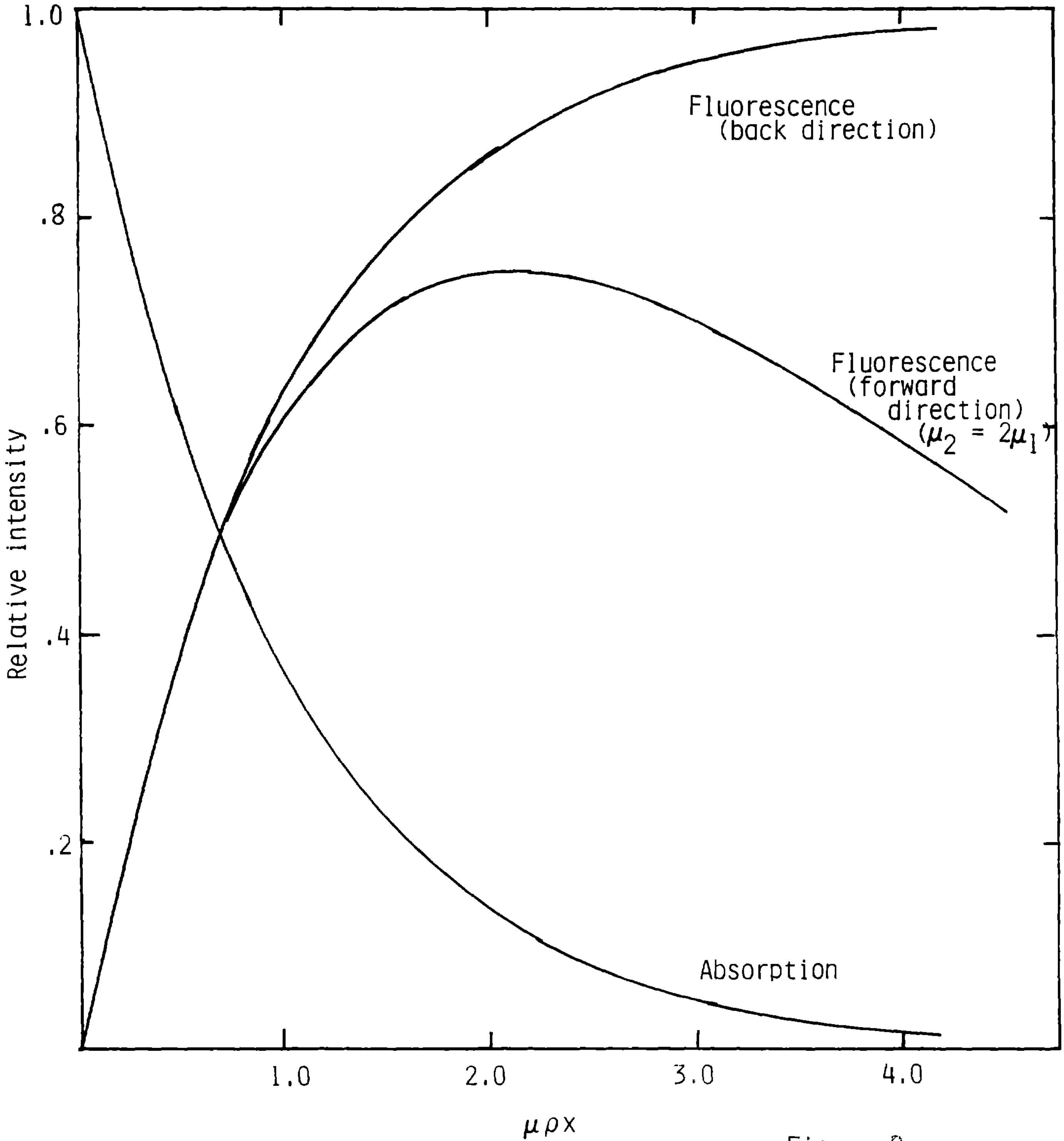


Figure 2

As a fraction:

$$\frac{d(\rho x)}{(\rho x)} = - \frac{1}{\mu\rho x} \cdot \frac{1}{N} \cdot dN$$

The error in the number of counts is $N^{1/2} \equiv \Delta N$:

$$\frac{\Delta(\rho x)}{(\rho x)} = - \frac{1}{\mu\rho x} \cdot \frac{1}{N^{1/2}}$$

$$= \frac{1}{\ln(^R/R_o)} \cdot \frac{1}{N^{1/2}} \quad .$$

We next seek the minimum in fractional error:

$$\frac{d}{dN}\left(\frac{\Delta(\rho x)}{\rho x}\right) \equiv 0 = \frac{d}{dN}\left\{ \frac{1}{N^{1/2}} \cdot \frac{1}{\ln(^R/R_o)} \right\}$$

$$= \frac{d}{dN}\left\{ \frac{1}{N^{1/2}} \cdot \frac{1}{\ln(N) - \ln(R_o t)} \right\}$$

$$0 = - \frac{1}{2} \cdot \frac{1}{\ln(^R/R_o) N^{3/2}} - \frac{1}{(\ln(^R/R_o))^2 \; N^{3/2}}$$

$$^R/R_o = e^{-2}$$

Therefore, the optimum measurement of areal density as measured by absorption is made when $\mu\rho x = 2$.

If we define the optimum range of thickness as that which limits the error to 2 times the minimum:

$$\sigma_{min} = -\frac{1}{2} \cdot \frac{1}{N^{1/2}}$$

$$2\sigma_{min} = -\frac{1}{N^{1/2}} = -\frac{1}{\mu\rho x'} \cdot \frac{1}{(N')^{1/2}}$$

$$N^{1/2} = (N')^{1/2}\ \mu\rho x'$$

$$\left(R_o te^{-\mu\rho x}\right)^{1/2} = \left(R_o te^{-\mu\rho x'}\right)^{1/2} \cdot \mu\rho x'$$

$$.3679 = \left(e^{-\mu\rho x'}\right)^{1/2} \cdot \mu\rho x'$$

By iterative guesses,

$\mu\rho x' = .4633$ and $5.357$

$R'/R_o = .629$ and $.00472$

2) Fluorescence, reverse direction

Basic eqn: $R/R_o = 1-e^{-\mu\rho x}$

The count rate is: $R = N/t$

so that: $\frac{N}{R_o t} = 1-e^{-\mu\rho x}$

Solving for areal density:

$$\rho x = -\frac{1}{\mu}\ln\left(1-\frac{N}{R_o t}\right)$$

Find the sensitivity of the areal density to changes in the number of counts:

$$\frac{d(\rho x)}{dN} = \frac{1}{\mu}\cdot\frac{1}{1-{}^{N}/R_o t}\cdot\frac{1}{R_o t}$$

$$= \frac{1}{\mu}\cdot\frac{1}{1-{}^{R}/R_o}\cdot\frac{R}{R_o N}$$

$$d(\rho x) = \frac{1}{\mu}\cdot\frac{1}{1-{}^{R}/R_o}\cdot\frac{{}^{R}/R_o}{N}\cdot dN$$

As a fraction: $\frac{d(\rho x)}{\rho x} = \frac{1}{\mu\rho x}\cdot\frac{1}{1-{}^{R}/R_o}\cdot\frac{{}^{R}/R_o}{N}\cdot dN$

The error in the number of counts is $N^{1/2} = \Delta N$ :

$$\frac{\Delta(\rho x)}{\rho x} = \frac{1}{N^{1/2}}\cdot\frac{1}{\mu\rho x}\cdot\frac{{}^{R}/R_o}{(1-{}^{R}/R_o)}$$

$$= \frac{-1}{N^{1/2}}\cdot\frac{1}{\ln(1-{}^{R}/R_o)}\cdot\frac{{}^{R}/R_o}{(1-{}^{R}/R_o)}$$

We next seek the minimum in the fractional error:

$$\frac{d}{dN}\left(\frac{\Delta(\rho x)}{\rho x}\right) \equiv 0 = \frac{d}{dN}\left\{\frac{-^{N}/R_o t}{N^{1/2} \cdot \ln\left(1-{}^{N}/R_o t\right)\left(1-{}^{N}/R_o t\right)}\right\}$$

Make the substitutions:

numerator = u

denominator = v

Recall that $d\,\frac{u}{v} = \frac{v\,du - \mu\,dv}{v^2}$

The differential will be zero when $v\,du - \mu dv = 0$

$$v\,du = \mu\,dv$$

$$N^{1/2} \cdot \ln\left(1 - {}^{R}/R_o\right)\left(1 - {}^{R}/R_o\right) \cdot \left({}^{-1}/R_o t\right) =$$

$$\left({}^{-R}/R_o\right)\left\{\frac{1}{2} \cdot N^{-1/2} \cdot \ln\left(1 - {}^{R}/R_o\right)\left(1 - {}^{R}/R_o\right) - N^{1/2} \ln\left(1 - {}^{R}/R_o\right)\left({}^{R}/R_o t\right) - \frac{N^{1/2}\left(1 - {}^{R}/R_o\right)\left({}^{1}/R_o t\right)}{1 - {}^{R}/R_o}\right\}$$

$$1 = \left\{\frac{R_o t}{2N} - \frac{1}{\left(1-{}^{R}/R_o\right)} - \frac{1}{\ln\left(1-{}^{R}/R_o\right)\left(1-{}^{R}/R_o\right)}\right\}{}^{R}/R_o$$

$$1 = \left\{\frac{1}{2\left({}^{R}/R_o\right)} - \frac{1}{\left(1-{}^{R}/R_o\right)} - \frac{1}{\ln\left(1-{}^{R}/R_o\right)\left(1-{}^{R}/R_o\right)}\right\}{}^{R}/R_o$$

$$1 = \frac{1}{2} - \frac{{}^R/R_o}{(1-{}^R/R_o)} - \frac{{}^R/R_o}{\ln(1-{}^R/R_o)(1-{}^R/R_o)}$$

$$\frac{1}{2} \cdot \frac{R_o}{R}\left(1 - \frac{1}{{}^R/R_o}\right) = -1 - \frac{1}{\ln(1-{}^R/R_o)}$$

$$\frac{1}{2} \cdot ({}^R/R_o - 1) + 1 = \frac{-1}{\ln(1-{}^R/R_o)}$$

$$\frac{1}{2}({}^R/R_o + 1) = \frac{1}{\mu\rho x}$$

$$u\rho x = \frac{2}{1 + {}^{R}o/_R}$$

$$= \frac{2}{1 + \frac{1}{1 - e^{-\mu\rho x}}}$$

By the method of iterative guesses

$\mu\rho x = .6438$ and ${}^R/R_o = .4747$

Alternately, a series approximation may be used for the exponential:

$$e^{-a} = 1 - a + \frac{a^2}{2!} - \frac{a^3}{3!} + \cdot \cdot \cdot$$

For $a = .64$, $e^{-a} = .527$

2nd order appx : $e^{-a} \sim .565$ (+7.2% error)

3rd order appx : $e^{-a} \sim .521$ (-1.1% error)

If the second order approximation is used:

$$\mu\rho x \sim \frac{2}{1 + \dfrac{1}{1 - 1 + \mu\rho x - \frac{1}{2}(\mu\rho x)^2}}$$

$$\mu\rho x \sim \frac{2\left(\mu\rho x - \frac{1}{2}(\mu\rho x)^2\right)}{\mu\rho x - \frac{1}{2}(\mu\rho x)^2 + 1}$$

Rearranging, $(\mu\rho x)^2 - 4(\mu\rho x) + 2 = 0$

$$\mu\rho x \sim .59 \quad \text{and} \quad {}^{R}/R_o \sim .46$$

The reader may show that the third order approximation gives a value of

$$\mu\rho x \sim .656 \quad \text{and} \quad {}^{R}/R_o \sim .481$$

We again seek the range of thicknesses over which the measurement error is limited to 2x the minimum:

$$\sigma_{min} = \frac{1}{N^{1/2}} \quad \frac{{}^{R}/R_o}{\ln(1-{}^{R}/R_o)\,(1-{}^{R}/R_o)} \quad ; \; {}^{R}/R_o = .4747$$

$$\sigma_{min} = \frac{-1.404}{N^{1/2}}$$

$$\sigma_{max} = \frac{-2.808}{(Rt)^{1/2}} = \frac{1}{(R't)^{1/2}} \cdot \frac{{}^{R'}/R_o}{\ln(1 - {}^{R'}/R_o)(1 - {}^{R'}/R_o)}$$

$$-4.076 = \frac{({}^{R'}/R_o)^{1/2}}{\ln(1 - {}^{R'}/R_o)(1 - {}^{R'}/R_o)}$$

By iterative guesses,

$\mu\rho x' = .06655$ and ${}^{R'}/R_o = .06438$

$\mu\rho x' = 2.286$ and ${}^{R'}/R_o = .8983$

3.) Fluorescence, forward direction

$$\text{Basic eqn: } R = \frac{KC}{\mu_1 - \mu_2} (e^{-\mu_2 \rho x} - e^{-\mu_1 \rho x})$$

The analysis will be limited to the thin side of the inflection point in the curve. First, let us find where that inflection point is. We find it by locating where the slope is zero:

$$\frac{dR}{d(\rho x)} = \frac{KC}{\mu_2 - \mu_1} \left\{ -\mu_2 e^{-\mu_2 \rho x} + \mu_1 e^{-\mu_1 \rho x} \right\} \equiv 0$$

$$\mu_2 e^{-\mu_2 \rho x} = \mu_1 e^{-\mu_1 \rho x}$$

$$\frac{\mu_2}{\mu_1} = e^{-(\mu_1 - \mu_2)\rho x}$$

$$\ln\left(\frac{\mu_2}{\mu_1}\right) = e^{-(\mu_1 - \mu_2)px}$$

Maximum is at:

$$(\rho x) = - \ln\left(\frac{\mu_2}{\mu_1}\right) \div (\mu_1 - \mu_2)$$

Let's look at some examples:

a) $\mu_2 \rightarrow \mu_1$, ie, $\mu_2 = \mu_1 + \Delta$

$$(\rho x) = - \ln (1 + {}^{\Delta}/\mu_1) \div (-\Delta)$$

$$= {}^{1}/\mu_1$$

$$\mu_1 \rho x = 1$$

$$R_{max} = \frac{KC}{-\Delta}\left(e^{-(\mu_1 + \Delta)\rho x} - e^{-\mu_1 \rho x}\right)$$

$$= + \frac{KC}{\Delta} e^{-\mu \rho x}(1 - \bar{e}^{\Delta\rho x})$$

$$= + \frac{KC}{\Delta} e^{-\mu_1 \rho x} (\Delta\rho x) = KC(\rho x)\, e^{-\mu_1 \rho x} = .368\ KC\ (\rho x)$$

$$R_o = \frac{KC}{\mu_1 + \mu_2} = \frac{KC}{2\mu_1}$$

$$\frac{R_{max}}{R_o} = .736$$

b) $(\mu_2) = 2\mu$ • This is approximately the case for gold fluorescence excited by Ag K$\alpha$

$$(\rho x) = -\ln(2) \div (-\mu_1) = \frac{.693}{\mu_1}$$

$$\mu_1 \rho x = .693$$

$$R_{max} = \frac{KC}{-\mu_1}\left(e^{-2\mu_1 \rho x} - e^{-\mu_1 \rho x}\right)$$

$$= \frac{KC}{\mu_1} e^{-\mu_1 \rho x}\left(1 - e^{-\mu_1 \rho x}\right)$$

$$= \frac{KC}{\mu_1} \times .25$$

$$R_o = \frac{KC}{\mu_1 + \mu_2} = \frac{KC}{3\mu_1}$$

$$\frac{R_{max}}{R_o} = .75$$

c) $\mu_2 >> \mu_1$

Let $\mu_2 = A\mu_1$, where A is a large number

$$(\rho x) = \frac{1}{A\mu_1} \ln A$$

$$\mu_1\rho x = \frac{1}{A} \ln A \quad ; \quad \mu_1\rho x = .26 \text{ when } A \sim 10$$

$$R_{max} = \frac{KC}{A\mu_1}\left(e^{-A\mu_1\rho x} - e^{-\mu_1\rho x}\right)$$

$$= \frac{KC}{A\mu_1} e^{-\mu_1\rho x}\left(1 - e^{(A - 1)\rho x}\right)$$

$$R_o = \frac{KC}{A\mu_1}$$

$$\frac{R_{max}}{R_o} = e^{-\mu_1\rho x} \quad \text{when } A >> 1$$

$$\frac{R_{max}}{R_o} \sim .79 \text{ when } A \sim 10$$

$$\sim 1 \text{ when } A \to \infty$$

We see from the foregoing examples that

$$R_{max} \sim 3/4\ R_o$$

where $R_{max}$ is the highest measurable count rate in the forward direction and $R_o$ is the highest (i.e., saturation) count rate in the back direction.

The range of values for $\mu_2$ is

$$\mu_1 < \mu_2 \lesssim 10\ \mu_1$$

Since we are limiting the analysis to the thin side of the inflection point, the examples show that the range of $(\mu_1 + \mu_2)\rho x$ is zero to about 2 to $\sim$ 2.8

We again seek the minimum error in the measurement of areal density.

The starting eqn is $R = \frac{KC}{\mu_1 - \mu_2}\left(e^{-\mu_2 \rho x} - e^{-\mu_1 \rho x}\right)$

The calibration factor is measured by fluorescence in the backware direction:

$$R_o = \frac{KC}{\mu_1 + \mu_2}$$

$$K = \frac{R_o(\mu_1 + \mu_2)}{C}$$

so that $\frac{R}{R_o} = \frac{(\mu_1 + \mu_2)}{(\mu_1 - \mu_2)}\left(e^{-\mu_2 \rho x}\ \ e^{-\mu_1 \rho x}\right) = \frac{N}{R_o t}$

In order to solve for the areal density, a series approximation or an iterative method must be used. With larger values for the exponential term, too many terms in the series approximation are required to be very useful, so an iterative method must be used. However, we already know from the back direction analysis that the optimum thickness is when $\mu\rho x \sim 0.6$. This value for the exponential is more or less adequately represented by a second order approximation (error < 6%).

$$\frac{R}{R_o} = \frac{\mu_1 + \mu_2}{\mu_1 - \mu_2} \left\{ 1 - \mu_2\rho x + \frac{1}{2} (\mu_2 \rho x)^2 - 1 + \mu_1 \rho x - \frac{1}{2} (\mu_1 \rho x)^2 \cdot \cdot \cdot \right\}$$

$$= \frac{\mu_1 + \mu_2}{\mu_1 - \mu_2} \left\{ (\mu_1 - \mu_2)\rho x - (\mu_1 - \mu_2)\ (\mu_1 + \mu_2)(\rho x)^2 - \cdot \cdot \cdot \right\}$$

$$= (\mu_1 + \mu_2)\ \rho x - \frac{1}{2} (\mu_1 + \mu_2)\ (\rho x)^2 - \cdot \cdot \cdot$$

$$(\rho x)^2 - \frac{2}{\mu_1 + \mu_2} (\rho x) + \frac{2R/R_o}{(\mu_1 + \mu_2)^2} + \cdot \cdot \cdot = 0$$

$$(\rho x) \approx \frac{1}{(\mu_1 + \mu_2)} \left\{ 1 - (1 - 2R/R_o)^{1/2} \right\}$$

This is exactly the same expression as for fluorescence in the backward direction, as may have been anticipated. Therefore, we can use the result from that analyses:

minimum error in $(\rho x)$ when $\mu\rho x \sim .64$

By using numerical examples, it is found empirically that:

minimum error in $(\rho x)$ is at $\mu\rho x = .52$ and range for doubling the error is $\mu\rho x = .067$ to $1.5$.

Graphs of relative errors follow.

THICKNESS MEASUREMENT BY ABSORPTION

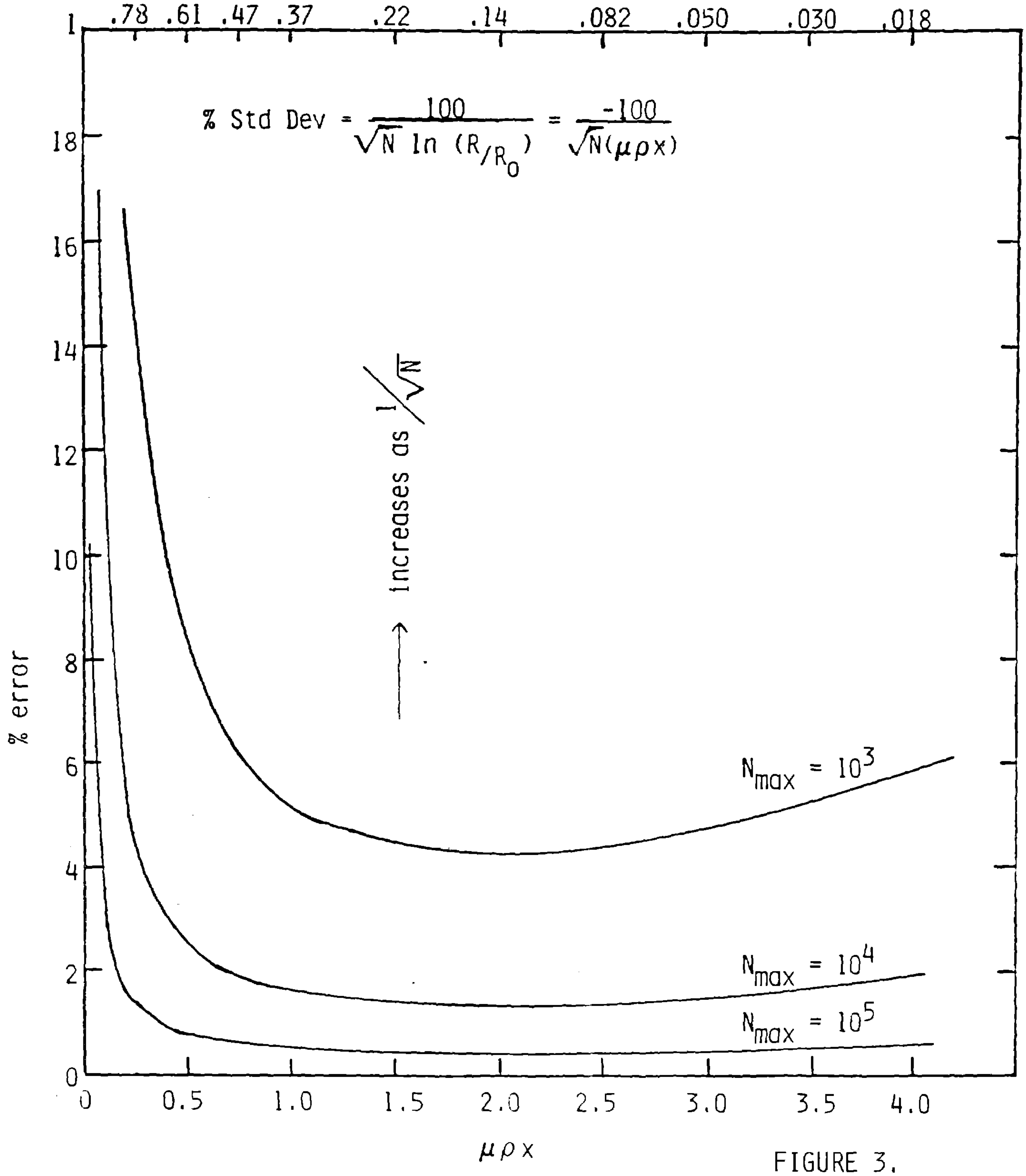


FIGURE 3.

THICKNESS MEASUREMENT BY FLUORESCENCE (Back-direction)

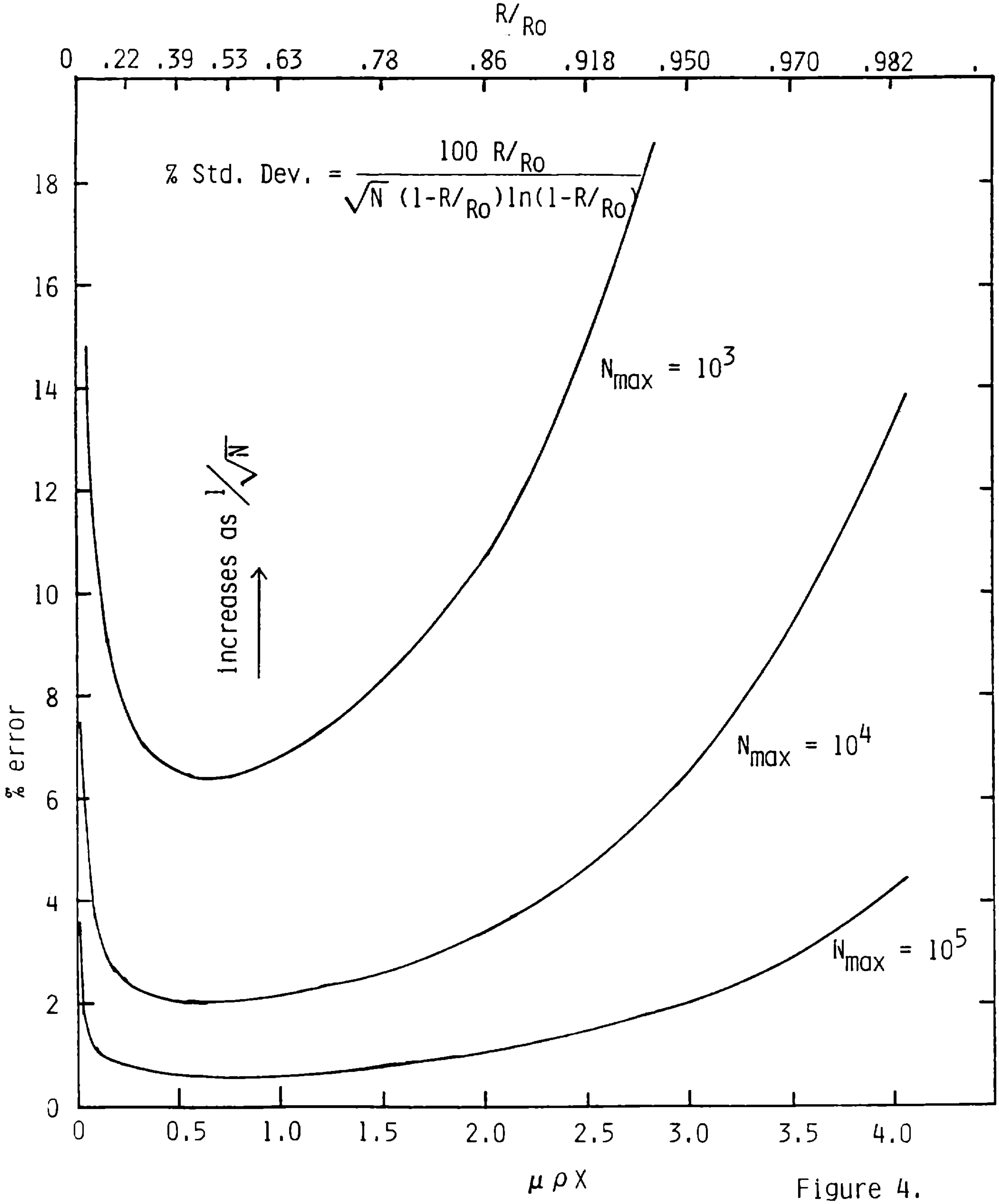


Figure 4.

THICKNESS MEASUREMENT BY FLUORESCENCE THROUGH FILM

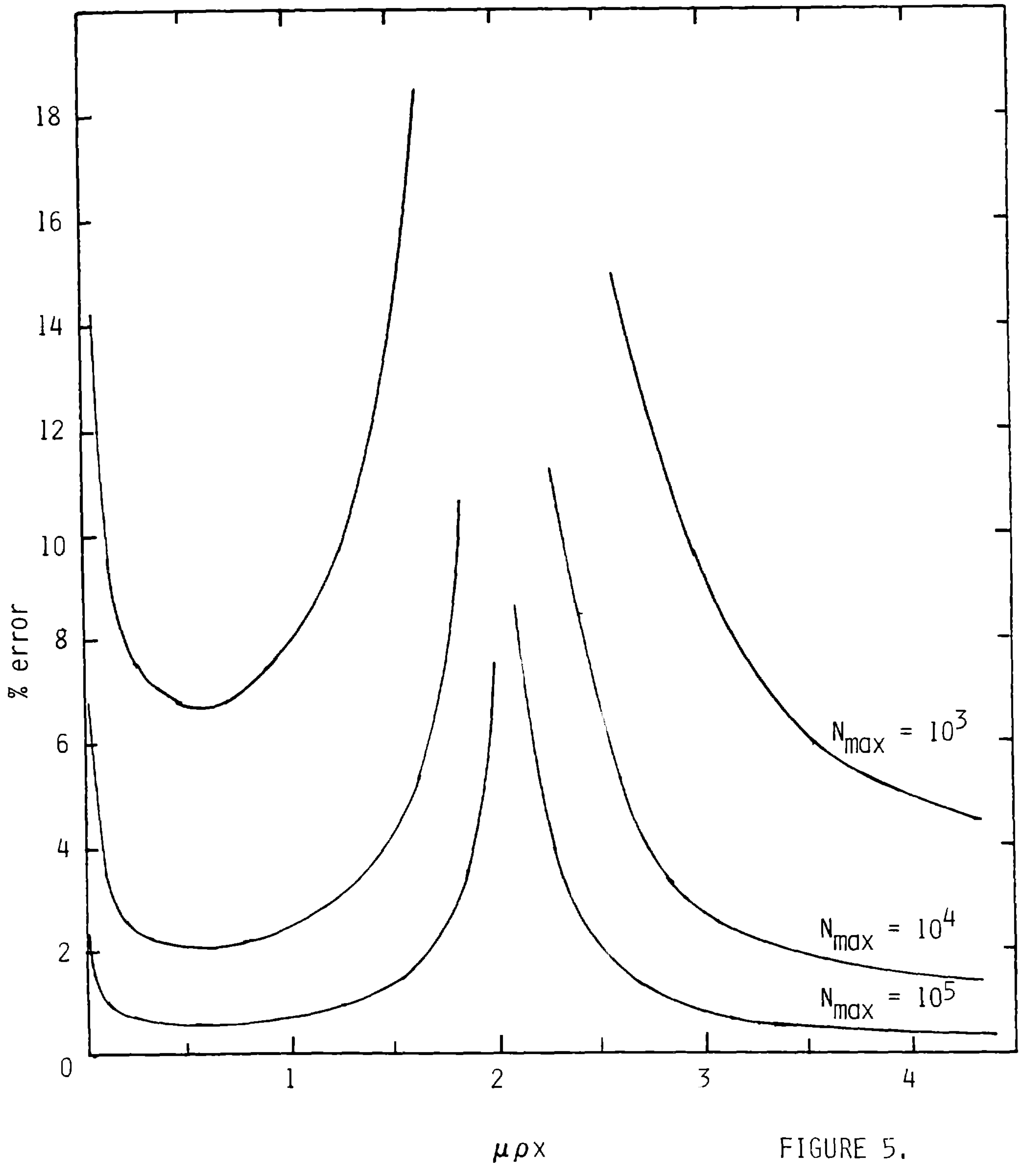


FIGURE 5.